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**DETERMINATION OF PLUTONIUM
CONCENTRATION AND DISTRIBUTION
IN URANIUM-PLUTONIUM MIXED OXIDE FUEL BY
AUTORADIOGRAPHY AND MICRODENSITOMETRY**

by

C. SARI

1971



Joint Nuclear Research Centre
Karlsruhe Establishment - Germany

European Institute for Transuranium Elements

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The dimensions of segregated particles emitting radiation and their distances can also be determined.

Examples of analysis performed on oxides, carbides and nitrides are reported.

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ABSTRACT

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KEYWORDS

QUANTITATIVE ANALYSIS
PLUTONIUM
PLUTONIUM ISOTOPES
PLUTONIUM OXIDES
PLUTONIUM NITRIDES
PLUTONIUM CARBIDES
PLUTONIUM COMPOUNDS
URANIUM ISOTOPES

URANIUM OXIDES
URANIUM NITRIDES
URANIUM CARBIDES
DENSITOMETERS
URANIUM COMPOUNDS
RADIOAUTOGRAPHY
RADIOISOTOPES
PHOTOGRAPHIC FILM

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Summary *)

The plutonium concentration and the homogeneity of the uranium-plutonium oxides, carbides and nitrides has been determined by microdensitometric analysis of autoradiographs of the examined radioactive material. The method is based on the fact that the blackening of a film is proportional to the concentration of the radiation emitted from the material. Correct measurements require the use of a reference material and standard curves which are independent of parameters such as exposure time of the film to the α radiations, the development time, inhomogeneities of the film and errors caused by wrong utilization of the microdensitometer.

Plutonium concentrations of less than 50 % in the solid can be determined with a precision of about 10 % and with about 5 % for higher concentrations. The microdensitometric measurements can be performed with an accuracy of ± 0.25 %. The aperture of the apparatus can be varied from a few μ^2 to a few mm^2 . The method allows the determination of the concentration of any material emitting radiation.

The dimensions of segregated particles emitting radiation and their distances can also be determined.

Examples of analyses performed on oxides, carbides and nitrides are reported.

Introduction

The production of metallic, ceramic or metalceramic uranium-plutonium compounds is performed by fusion or sintering processes. The sintering is essentially a heat treatment of pellets obtained by pressing mixed uranium-plutonium powders prepared by coprecipitation or by mechanical mixture. If this material is used as a nuclear fuel it is necessary, for security reasons (doppler effect, hot spots etc.), to know the distribution of the plutonium in the

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uranium matrix, and to determine the dimensions of the inhomogeneities.

There are three different analysis methods available now: α -counting, X-ray microanalysis, and autoradiography. Though the first two methods provide rather accurate measurements, they are not usually employed because they are time-consuming. The time required for a complete scanning of a specimen would be at least a few days.

The autoradiography carried out with normal [1], or colour [2] or cellulose nitrate [3] films is up to now the simplest and most useful method of analysis. It furnishes a total topography of the plutonium distribution and allows the identification of inhomogeneities and concentration profiles in the matrix. The quantitative autoradiographic determinations of materials emitting radiation were first developed in the biological field. The measurements were essentially based on the counting of radiation traces in the photographic emulsion with the help of a light microscope. This method was successfully employed later for the analysis of nuclear material containing an amount of plutonium up to a maximum of 0.8 % [4]. Another and more suitable way of quantitative autoradiographic analysis is based on photodensitometric measurements of the photographic emulsion exposed to the radiations. The formula for the plutonium concentration C_p is then:

$$C_p \% = \frac{2 - \log T}{\gamma \cdot T_e} \quad [5]$$

were T_e = exposure time, $2 - \log T$ = density of the autoradiograph, γ = an optical constant. In this formula C_p % is inaccurate and difficult to reproduce because of the errors in the determination of γ , of T_e and of the density.

The present investigation has been undertaken with the aim to determine the plutonium concentration and redistribution in plutonium bearing material using a comparative autoradiomicrodensitometric method. This avoids the difficulties and the errors related with the extremely sensitive conditions required for the measurements of absolute density.

1. Experimental

1.1. Autoradiography

The specimens of uranium-plutonium samples (carbides, nitrides, oxides) were mounted in a setting resin. A high quality finish surface was obtained using suitably modified metallographic techniques apt for the work with radioactive material in α -tight glove boxes [6]. In order to avoid the contamination of the exposed film with radioactive particles the specimens were completely decontaminated. This operation, carried out in glove boxes, involved ultrasonic cleaning and stripping of the sample surfaces with adhesive tape in order to remove loose contamination. Then the decontaminated specimen was transferred to a non contaminated glove box and mounted in a special container, with a low meltingpoint (48°C) alloy, fig.1. Fig.2 represents the sequence of the mounting operations. The autoradiographs were obtained by pressing nuclear track recording films on the polished surface of the specimen. The exposure times were between a few seconds and a few minutes. The α radiation of uranium compounds does not form an image with such short exposure times, while that of plutonium compounds produces well defined track lines.

A less boring and time consuming method was frequently used for routine analysis. It consisted of interposing a 6μ thick sheet of Hostafan between the specimen surface and the film. The device represented in fig.3 included a plastic sack connected to an α -tight glove box, and was provided with windows of 20 to 50 cm^2 . These windows were closed by sticking Hostafan sheets on to the plastic. From outside the box the film was pressed against the specimen surface through the Hostafan window. This operation took less than 30 minutes.

1.2. Microdensitometry

The autoradiographs were quantitatively analysed with a Joyce and Loebel automatic double-beam recording microdensitometer. Measurements were performed with aperture from $0.4\text{ mm} \times 2.4\text{ mm}$ to $1\mu \times 10\mu$.

2. Results and discussion

2.1. Standard curves

Seven specimens of uranium-plutonium mixed oxides with a plutonium content of 5, 10, 15, 25, 45, 50 and 70 % have been mounted together in araldit in order to take an autoradiograph for all specimens simultaneously. The exposure time varied between 10 and 90 seconds. The autoradiographs were then analysed with the microdensitometer and the results are represented in fig.4. In fig. 5a and 5b the differences in density of the exposed and unexposed area of the photographic emulsion, represented in arbitrary units, are plotted against plutonium concentration and time. Fig. 5b shows that small variations of exposure time influence remarkably the determination of the concentration. Moreover other errors caused by the variation of development time, the development solution, the quality of photographic emulsions etc. must also be taken into account. For these reasons it has been considered necessary to use standard curves (fig.6) which are independent of the above mentioned parameters. In fig.6 h and h_0 are height differences between the lines of non exposed and exposed areas of the film; h_0 is always related to the reference material which must be examined simultaneously with the unknown material. The h and h_0 values are obtained from the graphs of fig.4: each composition is considered in turn as a reference material. The use of these standard curves replaces the measurements of absolute densities: the plutonium concentration of the analysed specimen is determined by the curve of the reference material, fig.6 and by the ratio $h-h_0/h_0$ obtained from the unknown and the standard composition.

2.2. Determination of plutonium concentration

2.2.1. Plutonium concentration of solid solutions

Fig.7 shows the autoradiographs and the microdensitometric graphs of two specimens of coprecipitated uranium-plutonium mixed oxides. The specimen ①, used as reference, has been heated at 2000°C for 8 hours in order to improve the homogeneity of the solid solution. For each sample the height

variation of the microdensitometric lines is proportional to the variation of plutonium content. In the standard curves of fig.6 the ratio $h-h_0/h_0$, calculated from the fig.7, corresponds to a value $Pu/(U+Pu) = 0.45$ which represents the plutonium concentration of the specimen (2).

The same procedure was used for carbides and nitrides: the ratios $h-h_0/h_0$ calculated from the graphs of fig.8 correspond respectively to $Pu/(U+Pu) = 0.28$ and $Pu/(U+Pu) = 0.19$.

2.2.2. Mean plutonium concentration of mechanical mixtures

The procedure of mechanical mixing of plutonium and uranium oxides is at present the only economic way to produce oxide fuel. It is therefore interesting to have a quick method of analysis for the determination of the mean plutonium concentration independent of the strong local variations.

The autoradiograph of fig.9 gives a picture of the distribution of plutonium oxide particles in a uranium oxide matrix. Each white spot in fig.9 represents roughly the area of a plutonium-rich particle. The microdensitometric determination of the mean plutonium concentration is performed as explained in 2.2.1. The only difference is the necessity to use a large aperture so that a sufficient number of spots is always present in the aperture area. If the dispersion of plutonium-rich-particles is homogeneous, rather accurate concentration measurements can be performed, otherwise (but this case is unusual) the results can be represented by histograms. The plutonium concentration of the specimen of fig.9 and 10 amounts to 15 %.

2.2.3. Determination of concentration profiles

The phenomena of diffusion or redistribution of material are always connected with concentration profiles. If the diffusing material is radioactive the autoradiographic and microdensitometric techniques provide accurate measurements in a very short time compared to the time required for α -counting or X-ray microanalysis procedures. In fig.11 and 11 bis the plutonium concentration profiles of specimens heated in a thermal gradient are shown. In both specimens a

plutonium redistribution occurred as a function of thermodiffusion and preferential evaporation processes [7, 8]. The trend of the curves obtained by autoradiomicrodensitometry and by α -counting are comparable, and the values are in good agreement. A striking difference is to be found in the time necessary for the measurements. In addition even if the window of the α -counter is relatively large (0.1×1 mm) only 150 α /min. pass through and therefore an accurate linear scanning of 1 cm of a surface requires about 90 hours. The autoradiomicrodensitometric measurement of the same 1 cm line takes less than 30 minutes. Moreover, the microdensitometer has a smaller aperture width and therefore better resolution.

Fig.12 shows the plutonium concentration profile in $(U_{0.30}Pu_{0.70})O_2$ mixed oxide spheres prepared by sol-gel. The growth of the particles occurred irregularly by superposition of layers with different plutonium content.

2.3. Evaluation of the dimensions of segregated plutonium-rich particles in a matrix and of their distances

Fig.13 represents the autoradiograph and the microdensitometric graph of a specimen prepared by mechanical mixture of $(U_{0.85}Pu_{0.15})O_2$ spheres in a uranium oxide matrix. Microdensitometric scanning of the autoradiograph provides information about the mean radius of the spheres and the mean distance between the dispersed particles. The radius was determined by the formula $R = r - 25$ where r is the half width of the peaks measured on the microdensitometric graphs and 25 is the range, in μ , of the α radiations recorded in the photographic emulsion [1]. According to the assumption of [1], an autoradiographic spot or a microdensitometric peak should always have a diameter greater than 50μ .

The data obtained from fig.12 and 13 are given in table I. Fig.14 represents the autoradiograph, the micrograph and the microdensitometric graph of an inhomogeneous solid solution. The inhomogeneities still present in the matrix after a heat treatment of 4 hours at $1650^\circ C$ are caused by an imperfect coprecipitation. The formula $R = r - 25$ cannot, in this case, provide accurate values because the segregated particles are

too small, too close to each other and interact. The value for an α -range of 25μ [1] is not realistic if the segregated particles are smaller than 10 microns. In this case only the metallographic analysis is helpful for quantitative determinations. Fig.14b represents the surface of an inhomogeneous specimen: some segregated particles are isolated from the matrix by selective etching procedures [9]. The mean dimensions of the particles are measured with the usual procedures of quantitative metallography (table I).

The distance between two particles is determined by the following formula $L = l - (R_1 + R_2)$ where l is the distance between two peaks of the microdensitometric graphs, $R_1 + R_2$ are the radii of the particles. The results of the measurements performed on specimens 12, 13 and 14 are given in table II. If the radii of the segregated particles are smaller than 10μ the values are incorrect for the reason explained above.

3. Conclusion

It seems, according to our experiments, that the autoradio-microdensitometric method best suit the necessity of correct and quick determination of plutonium concentration and plutonium redistribution, especially with regard to the fabrication processes. The use of standard curves makes the measurements easy and the use of absolute density values is avoided.

The reasons for using a comparative method are as follows:

- the density limits depend on a number of conditions such as the optical magnification, the aperture width and area,
- for a much smaller aperture the density limits are extended, if the optical magnification is reduced,
- for a very small aperture-width the density limit is lower than for a relatively large one,
- in the case of a detail having a size of the order of a

few microns or if the density is greater than 2,7 D, the aperture width must be chosen carefully,

- density values are only accurate if the microdensitometric scanning is made under identical conditions.

The method can be used in any range of concentration.

The dimensions of the plutonium-rich-particles, dispersed in the matrix, are determined by using the geometry of the sphere and assuming that a random assembly of these particles is cut by a plane section. The values are in general smaller than the true one's and the error increases with the decrease of the dimensions of the particles. If for instance the dispersed particles have a diameter smaller than 10μ a high proportion of them will be cut by the top or the bottom of the section of the fuel recording on the film (the depth of the section is about 15μ and corresponds to the range of the α radiations in the fuel material). This would give crude informations about the particles size range and therefore represents the limit of the method.

Table I

Autoradiomicrodensitometric and metallographic measurements of the diameter of the dispersed radioactive particles

Diameter, microns	
Autoradiomicrodensitometric techniques	Metallographic techniques
650	600
650	600
580	500
220	200
230	200
170	220
130	100
100	80
75	120
80	70
-	15
-	12
-	9

Table II

Autoradiometric and metallographic measurements
of the distances between dispersed radioactive
particles

Distances, microns	
Autoradiomicrodensito- metric techniques	Metallographic techniques
1400	1500
350	430
230	300
200	250
150	220
-	15
-	8
-	~1

Acknowledgements

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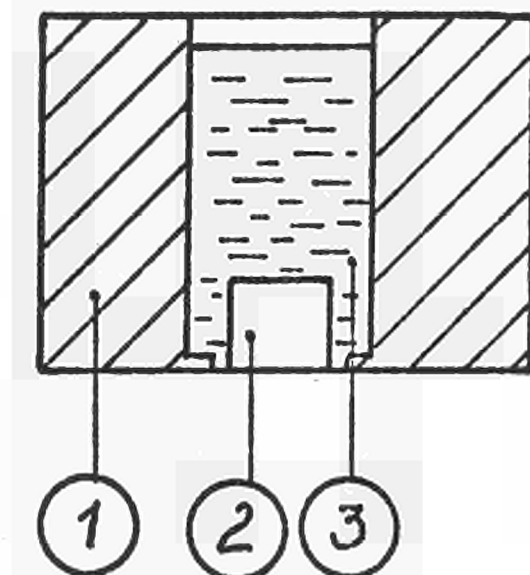


Fig. 1 Decontaminated plutonium compound to be handled out of glove box. (1) holder; (2) specimen; (3) low melting-point mounting alloy.

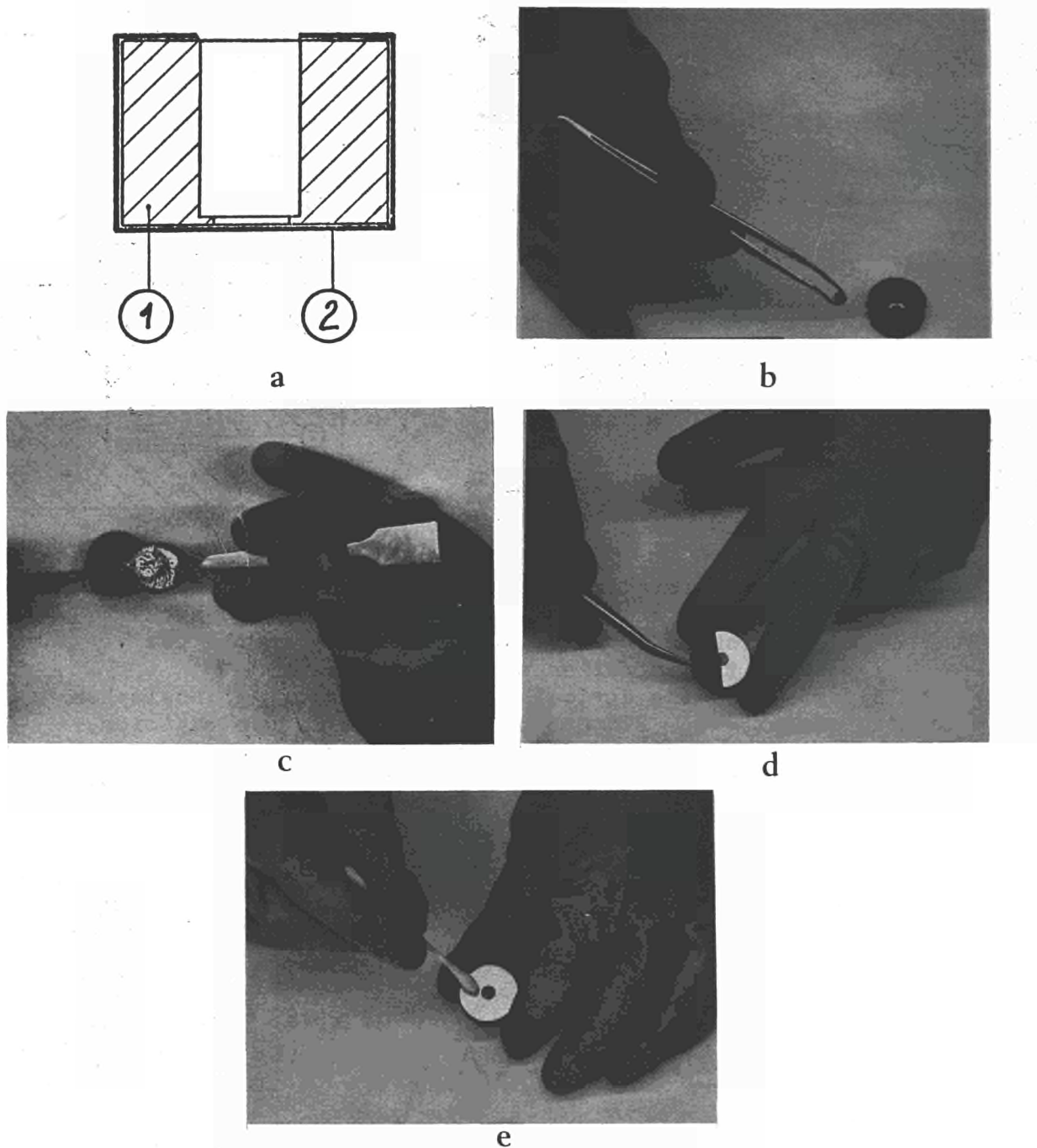


Fig. 2 Mounting sequences of a decontaminated specimen,
a) holder, ①, covered with adhesive tape; ② the tape prevents contamination of the holder. The top of the central hole is free.
b) introduction of the specimen in the holder hole
c) pouring the metallic mounting material at 60°C
d) removing the adhesive tape from the holder. At the center the decontaminated surface of the specimen.
e) wisck tests on the decontaminated surface of the specimen in order to determine the amount of loose contamination (a loose contamination of $10^{-5} \mu\text{C}/\text{cm}^2$ is allowed).

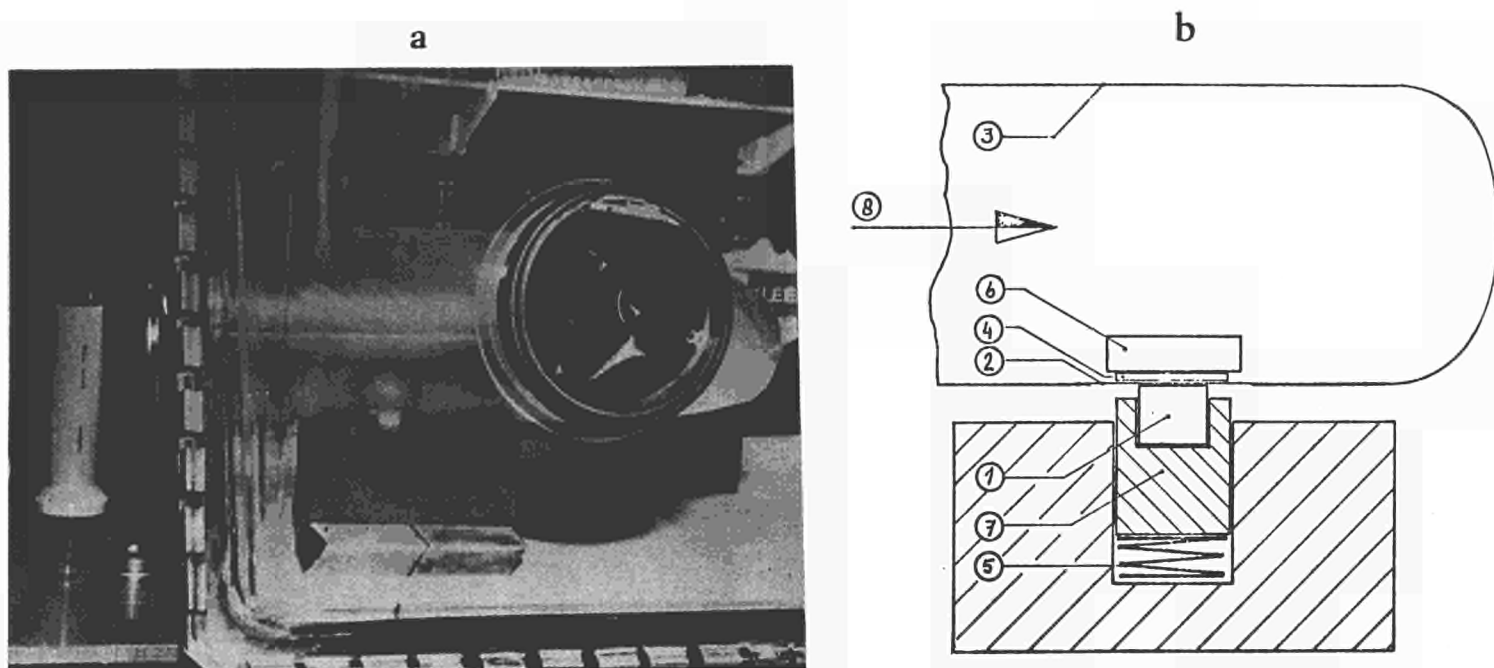


Fig. 3 Device for autoradiography

- a) side of the glove box connected with a PVC sack.
A depression of 20 mm water existing in the box causes the inflation of the sack.
- b) detail of the device: ① specimen, ② Hostaphan window, ③ PVC sack, ④ photographic emulsion, ⑤ spring, ⑥ load, ⑦ teflon support of the specimen, ⑧ to the non-contaminated side of the sack

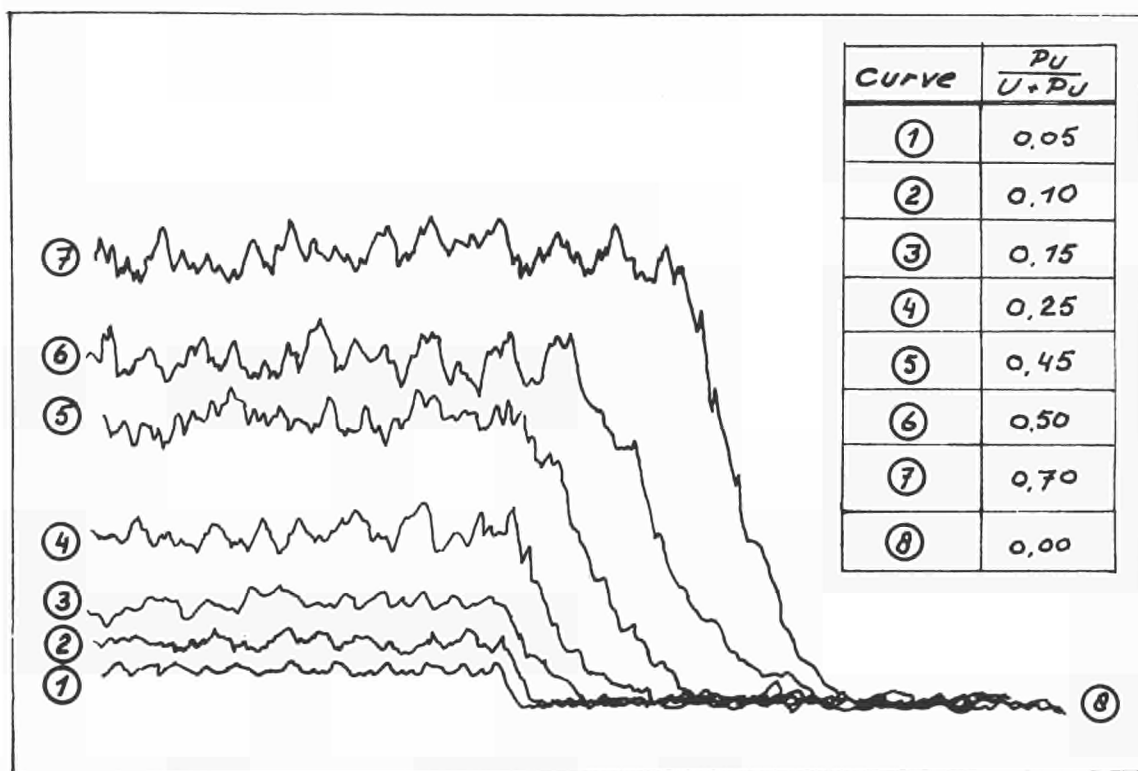


Fig. 4 Example of microdensitometric graphs obtained from autoradiographs of mixed oxide specimen exposed for 20 seconds. $Pu/U+Pu = 0.05, 0.10, 0.15, 0.25, 0.45, 0.5, 0.7$. Aperture $1.5 \times 100 \mu$. Optical magnification $\times 44$. Feedback setting ratio 1:200. Wedge range 0 - 1.5 D.

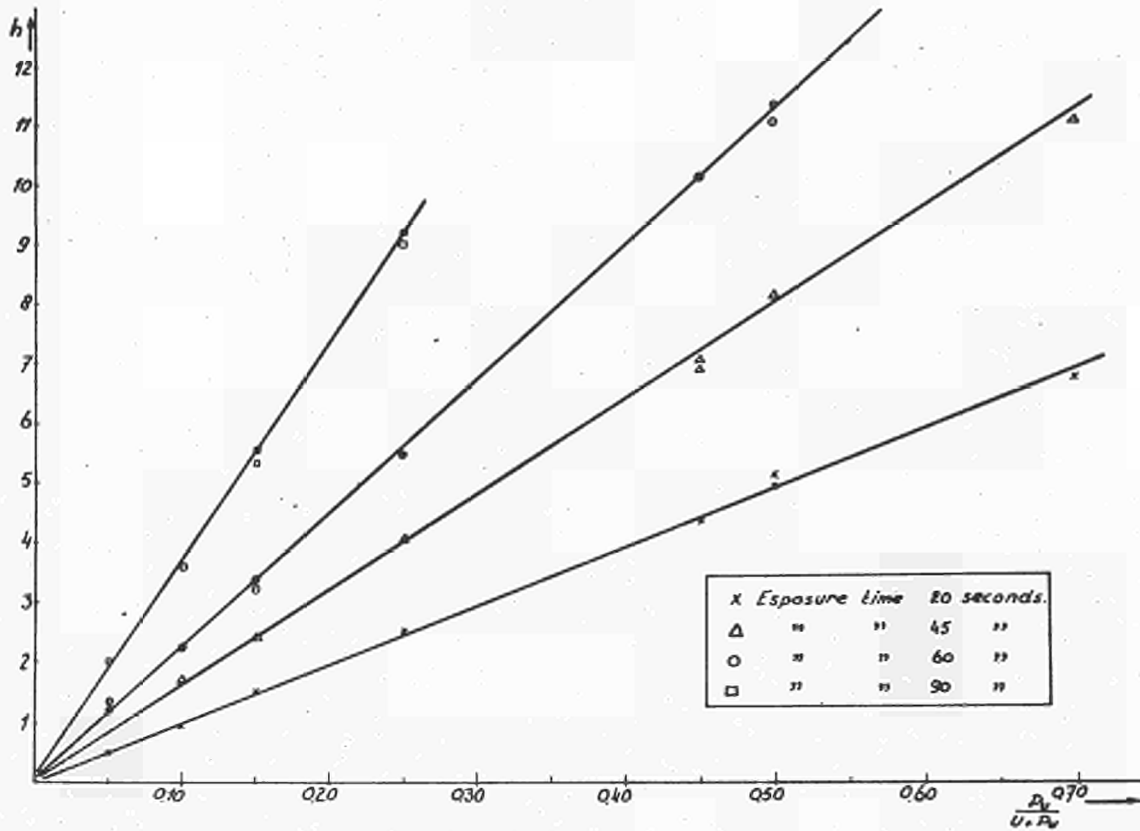


Fig. 5a Density of the autoradiographs, represented in arbitrary units, plotted against plutonium concentration.

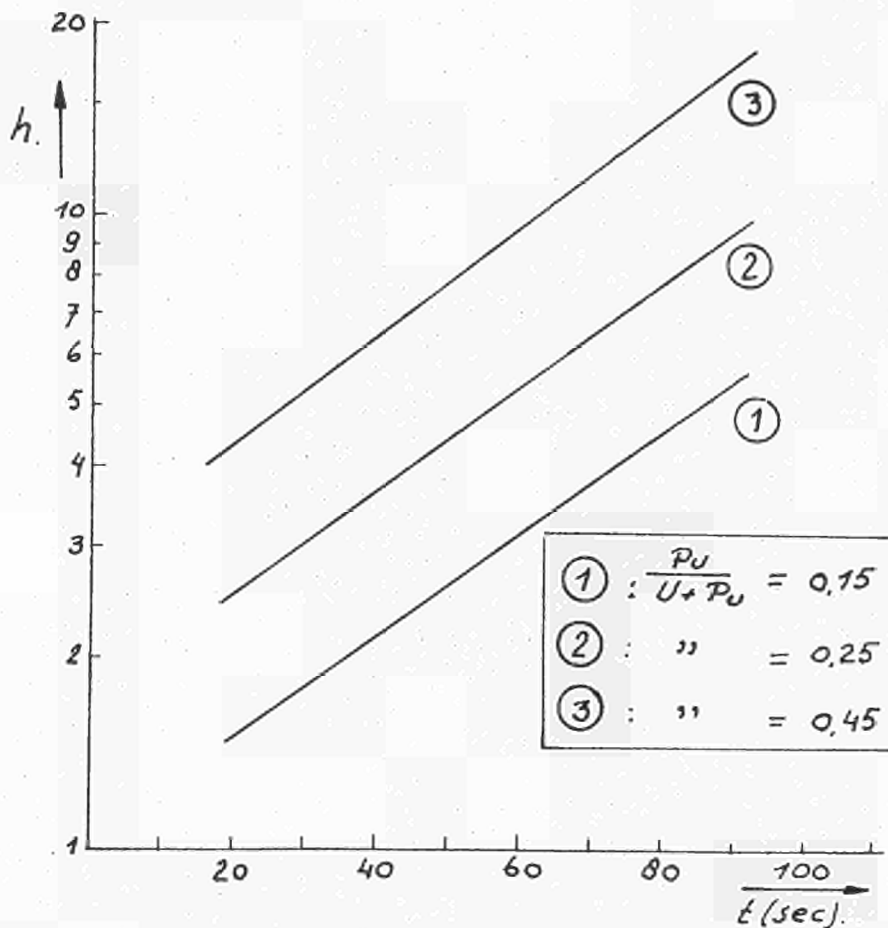


Fig. 5b Density of the autoradiographs, represented in arbitrary units, plotted against time.

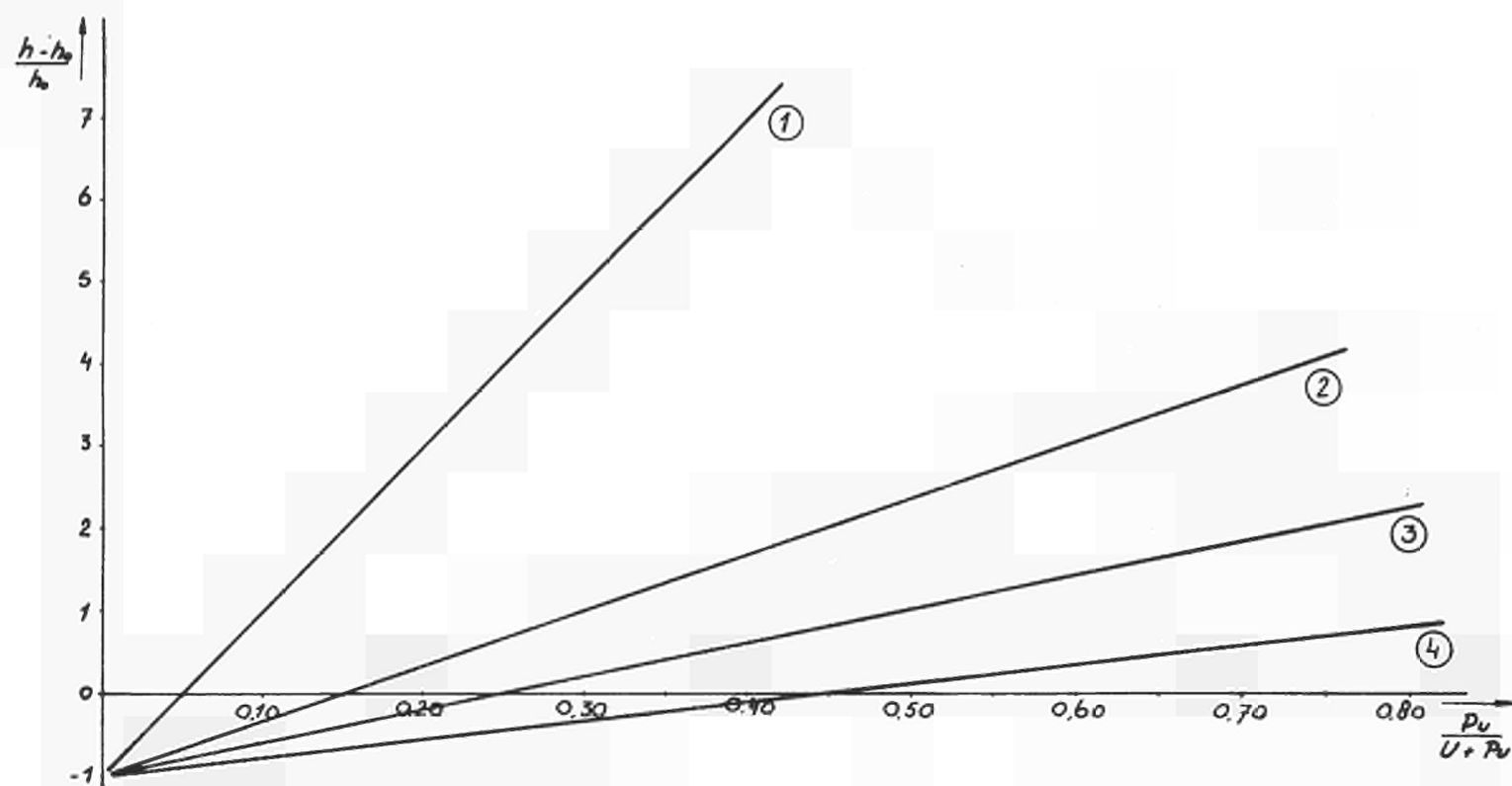


Fig. 6

Standard curves used for the determination of plutonium concentration in oxides, nitrides and carbides. ①, ②, ③, and ④ represent the curve obtained if the reference material has a composition $P_u/U+P_u$ respectively, of 0.05, 0.15, 0.25 and 0.45.

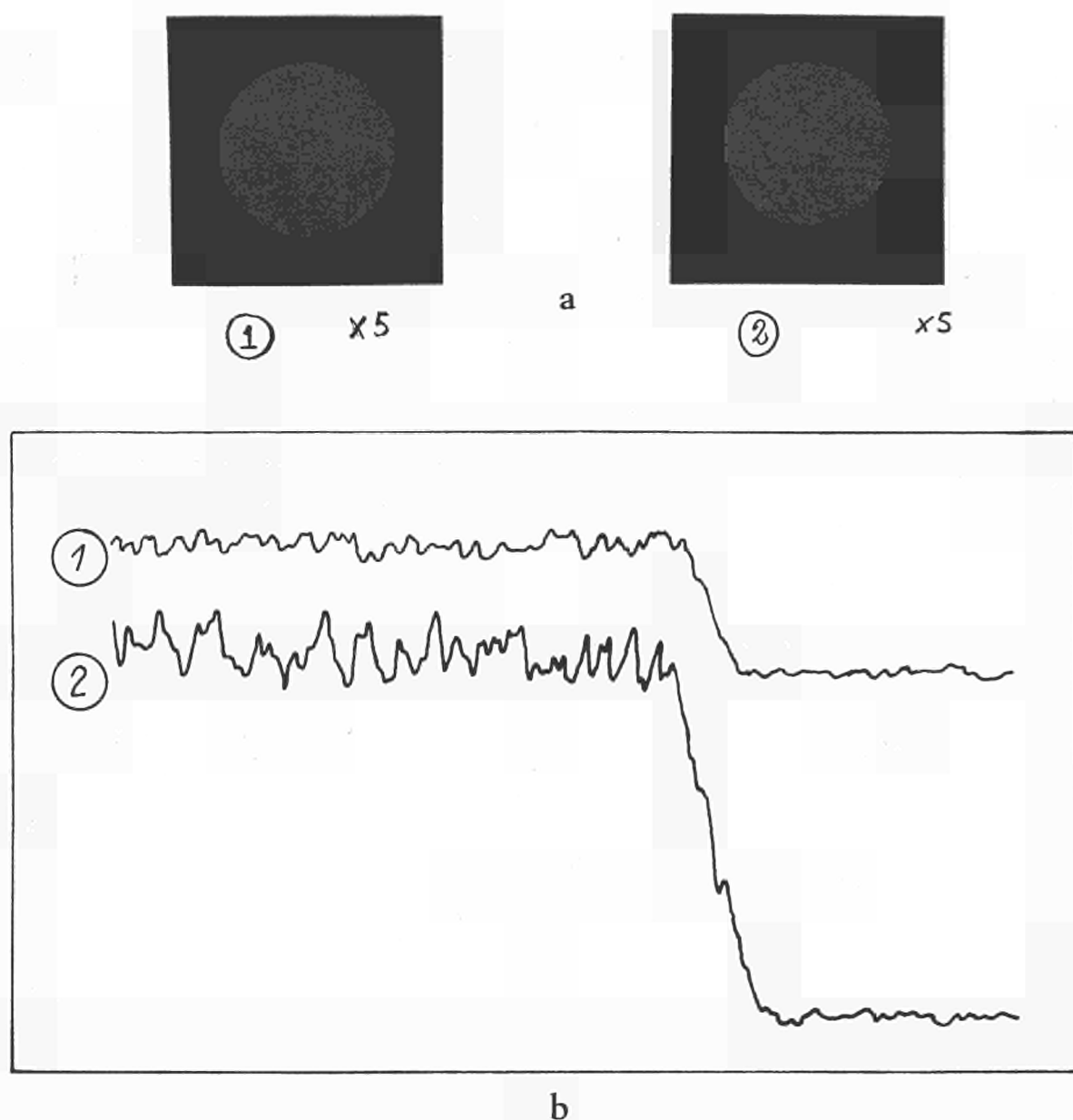


Fig. 7

a) autoradiographs of two oxide specimens exposed simultaneously, (1) is the reference material $(U_{0.85}Pu_{0.15})O_2$ and (2) is the specimen with unknown plutonium content. x 5
b) microdensitometric graphs of the specimens (1) and (2)

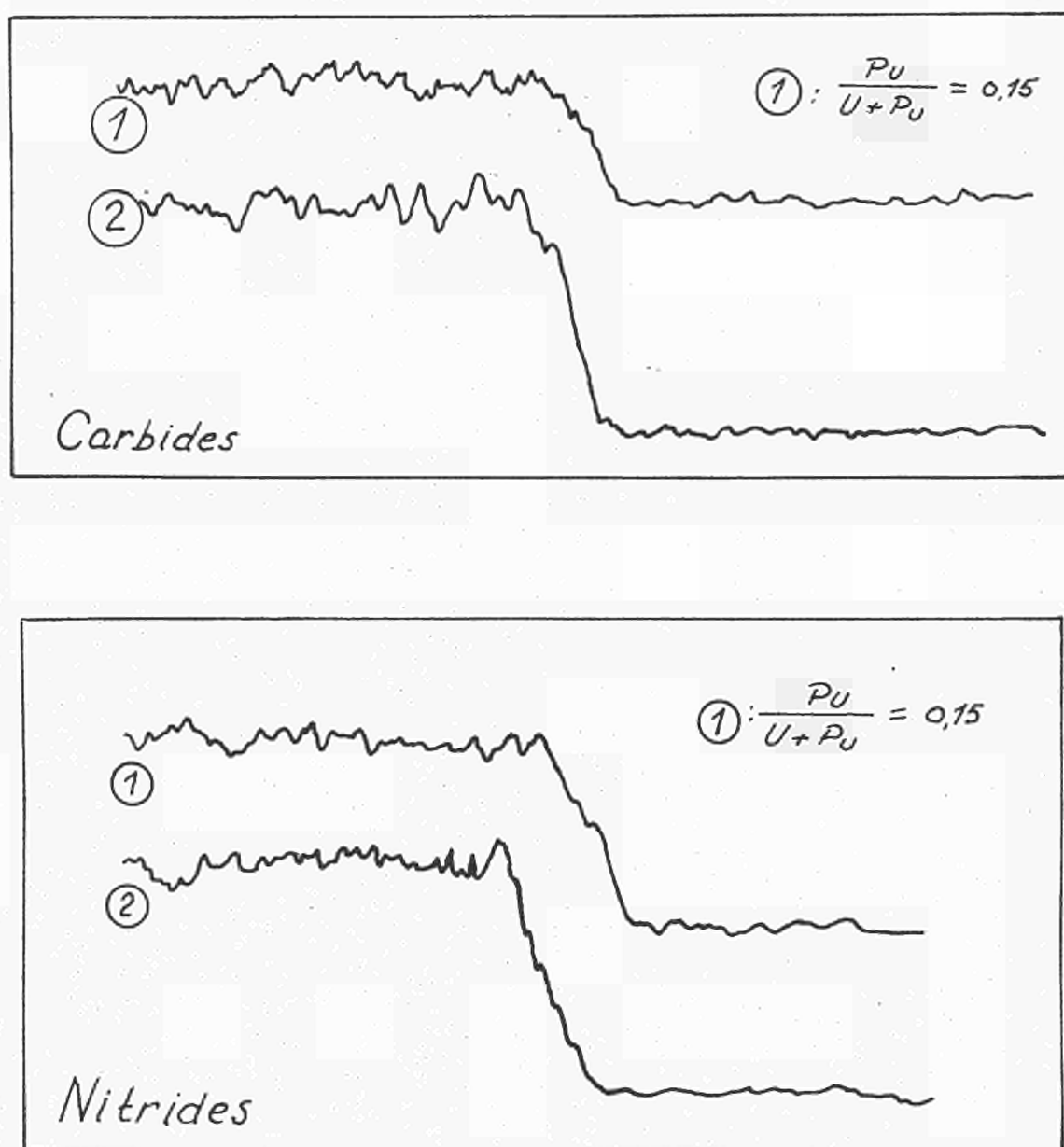
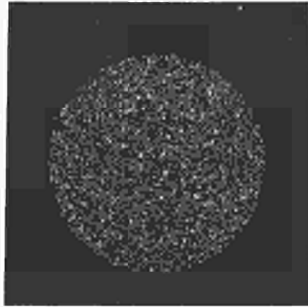


Fig. 8

Microdensitometric scanning of autoradiographs of carbide and nitride specimens. ① reference material and ② specimen with unknown plutonium content.



x5

Fig. 9 Autoradiograph of a mechanical mixture $\text{UO}_2\text{-PuO}_2$ white spots are plutonium-rich-particles. x5

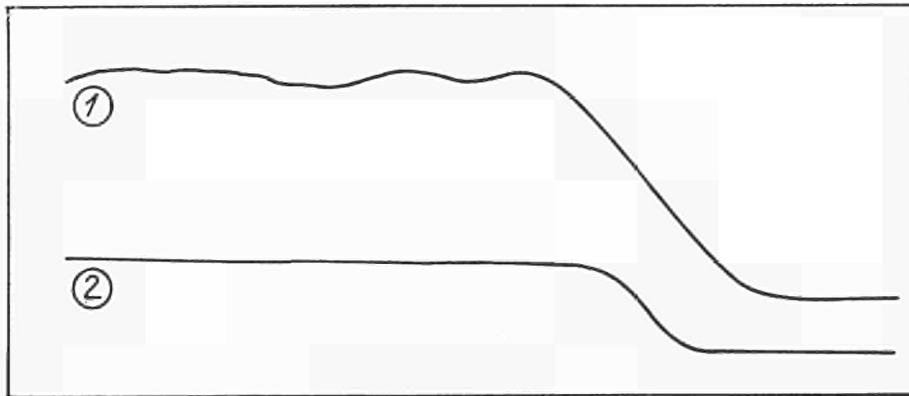


Fig. 10

① microdensitometric graph of the autoradiograph of Fig. 9

② microdensitometric graph of the autoradiograph of a reference material with composition $(\text{U}_{0.95}\text{Pu}_{0.05})\text{O}_2$.

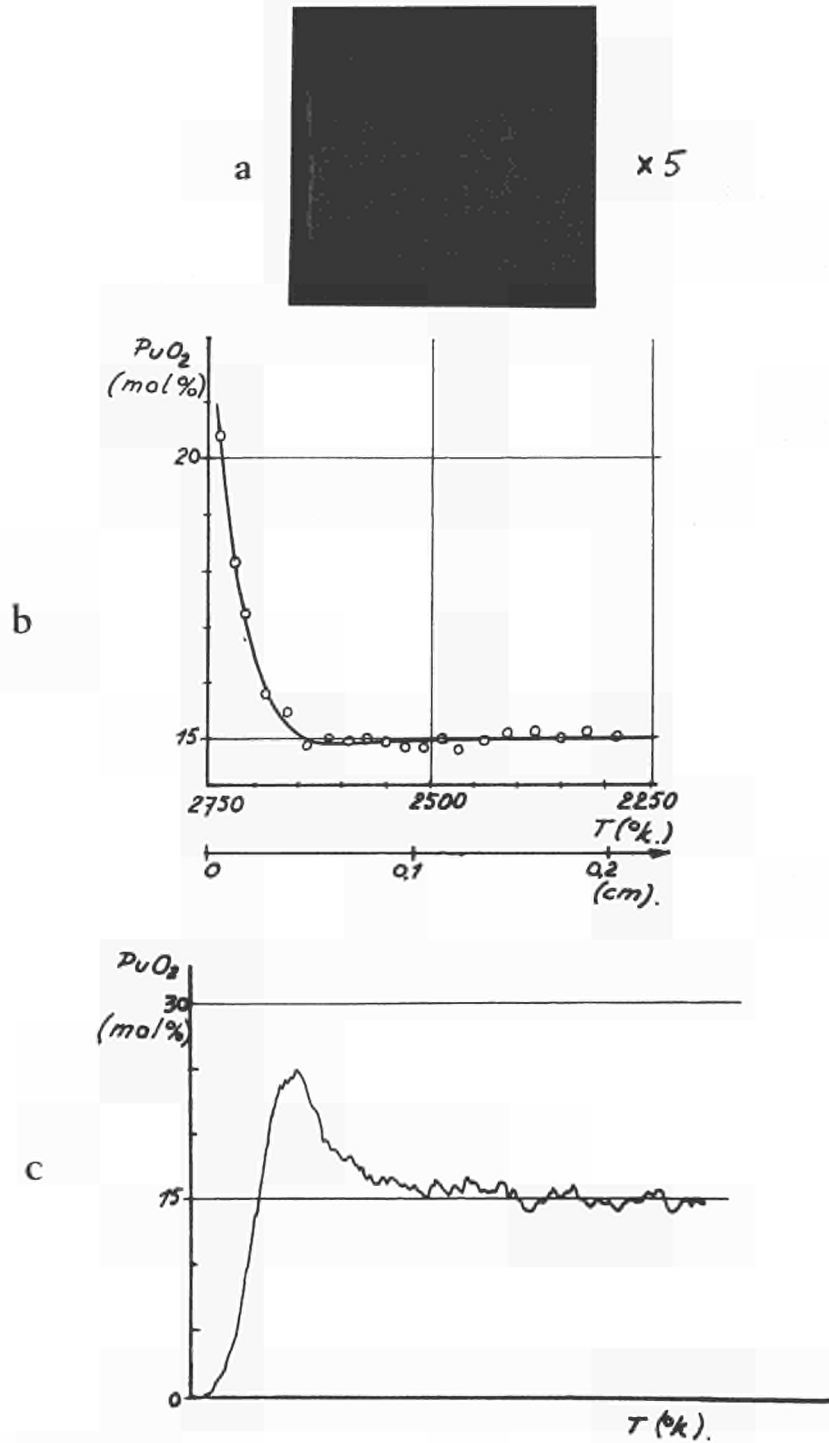


Fig. 11

Plutonium concentration profile of coprecipitated mixed oxide specimen $(\text{U}_{0.85}\text{Pu}_{0.15})\text{O}_2$ heated in a thermal gradient [7].

a) autoradiograph $\times 5$

b) concentration profile measured by α -counting techniques. Aperture 0.1×1 mm

c) concentration profile measured by autoradiomicrodensitometry. Aperture $1.5 \times 100 \mu$.

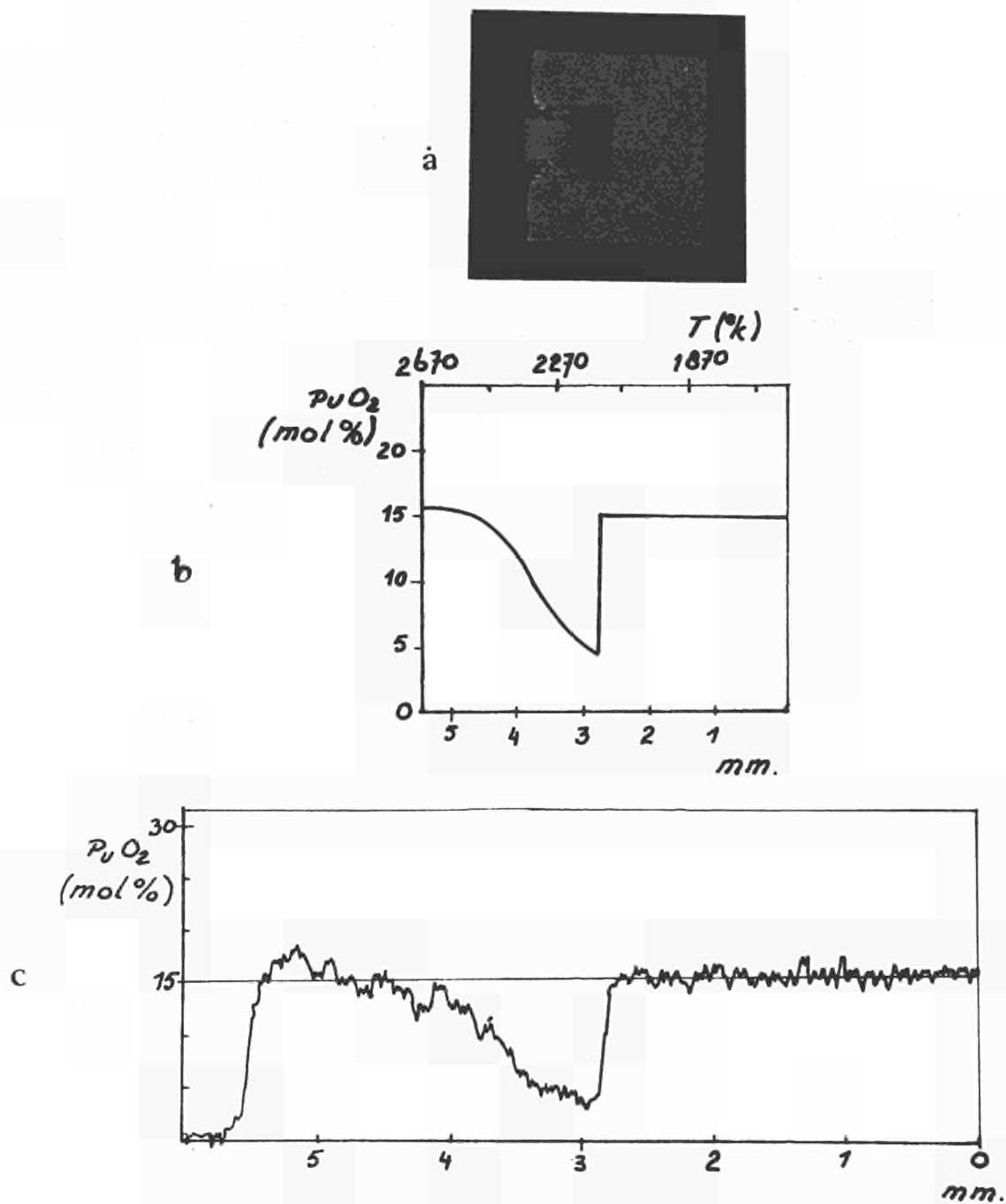


Fig. 11 bis Plutonium concentration profile of coprecipitated mixed oxide specimen $(U_{0.85}Pu_{0.15})O_2$ provided of a central well and heated in a thermal gradient [8].

a) autoradiograph x 5

b) concentration profile measured by α -counting techniques. Aperture 0.1 x 1 mm.

c) concentration profile measured by autoradiomicrodensitometry. Aperture 1.5 x 100 μ

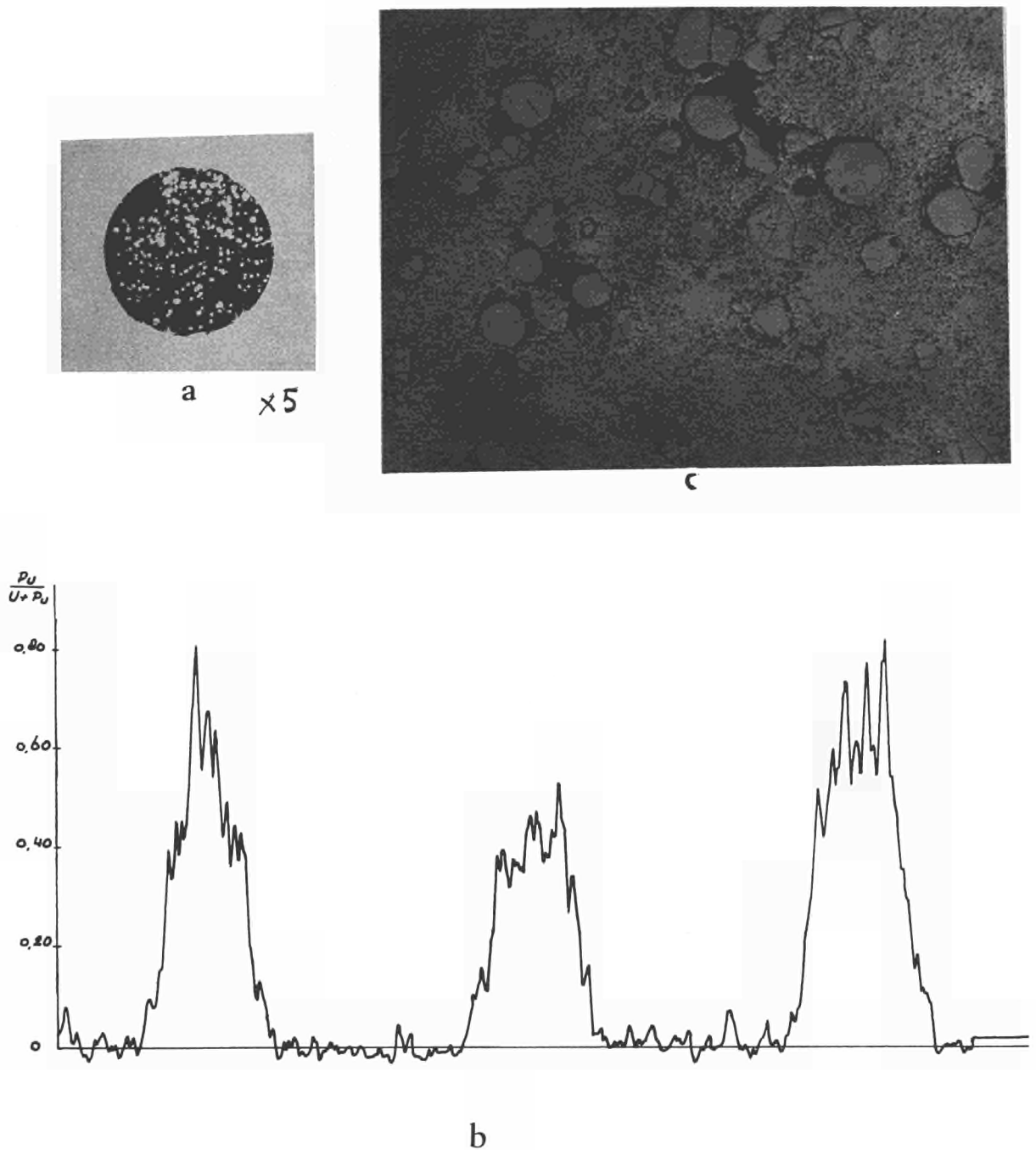


Fig. 12

Mechanical mixture of UO_2 powder and $(U_{0.30}Pu_{0.70})O_2$ spheres prepared by sol-gel.

a) autoradiograph, white spots represent the plutonium-bearing-particles. x 5

b) microdensitometric graph. Aperture 1.5×10^{-4} u. Optical magnification x 44. Feedback setting ratio 1 : 200

c) micrograph, roundshaped inclusions represent the plutonium-rich particles. x 50

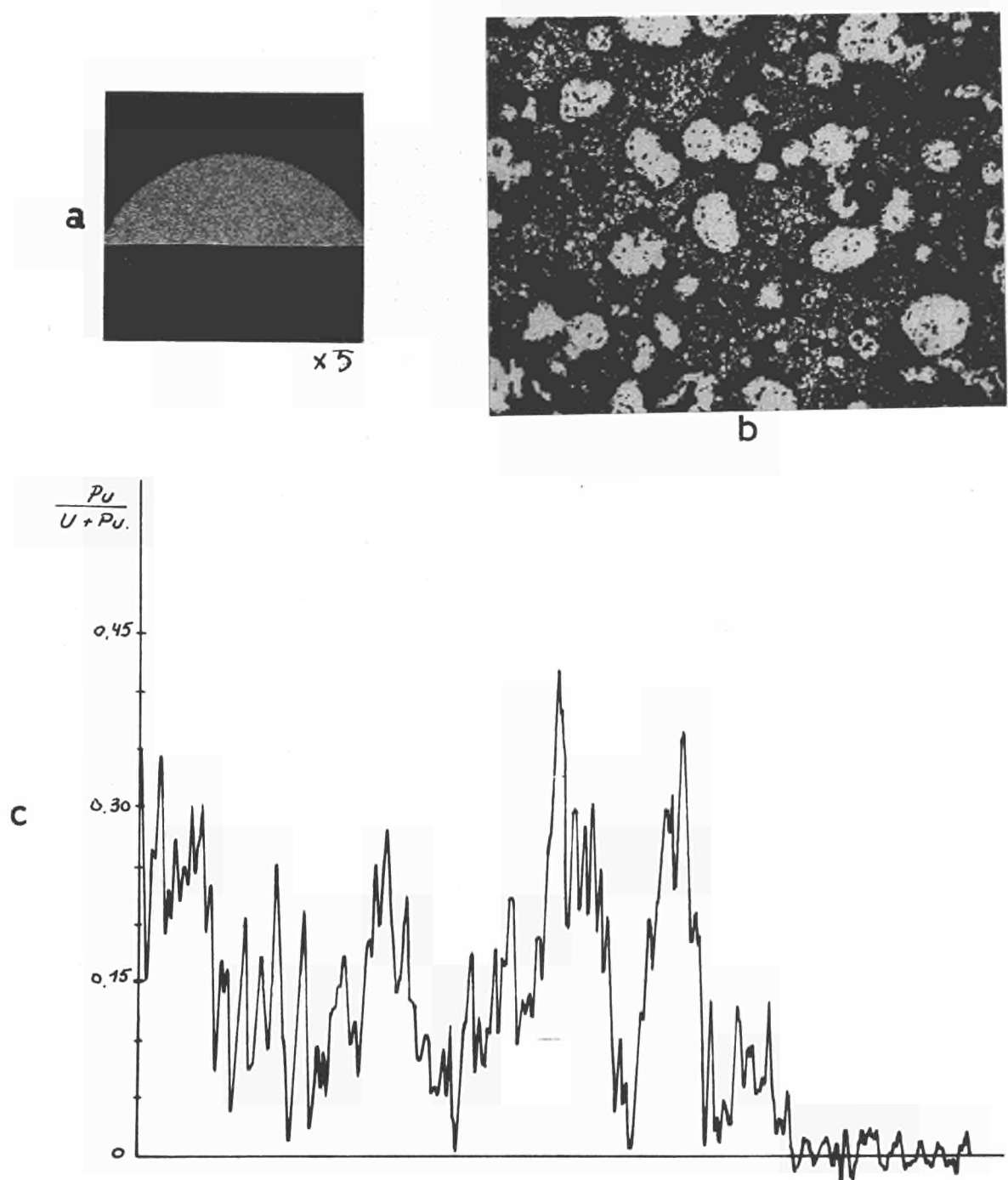


Fig. 14

Coprecipitated $(U_{0.85}Pu_{0.15})O_2$ mixed oxide. Inhomogeneous.

a) autoradiograph, white spots are plutonium-rich-particles, x 5

b) micrograph, white regions are plutonium-rich particles revealed by selective etching [9] x 800

c) microdensitometric graph of 14a. Aperture $1.5 \times 10^{-4} \mu$. Magnification x 44. Feedback setting ratio 1 : 200.

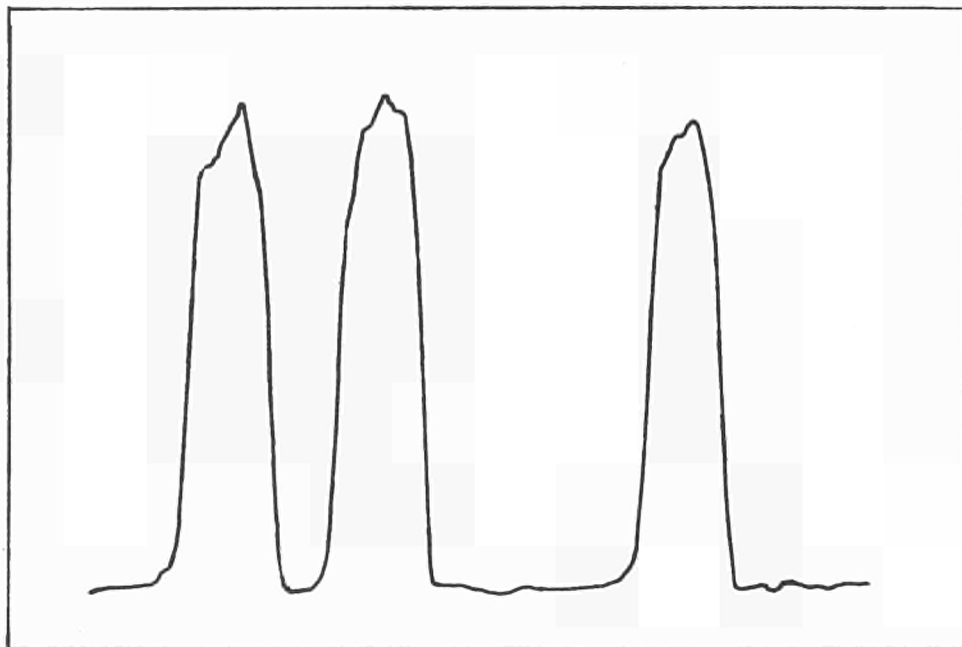
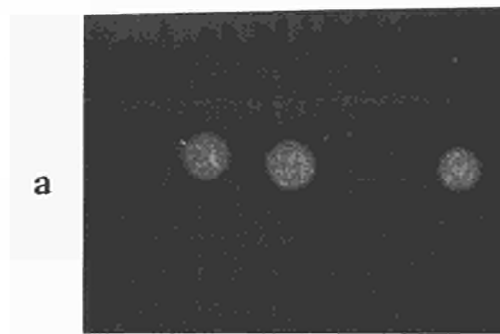


Fig. 13

Mechanical mixture of UO_2 powder and $(\text{U}_{0.85}\text{Pu}_{0.15})\text{O}_2$ spheres.

a) autoradiograph x 10

b) microdensitometric graph. Aperture $30 \times 1000 \mu$. Feedback setting ratio 1:20. Each peak represents a sphere of fig. 13a

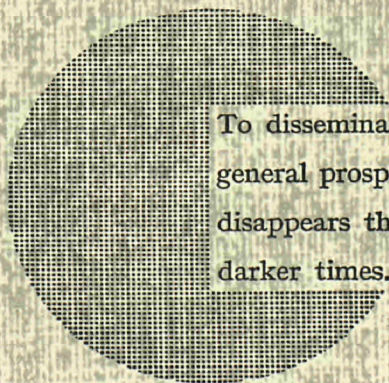
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Alfred Nobel

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